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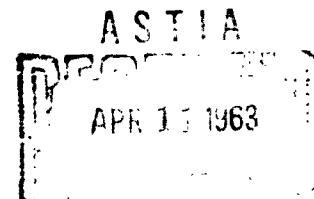
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## A Portfolio of Experience in Refractory Metal Protective Systems Research

11 MARCH 1963

*Prepared by D. H. LEEDS  
Materials Sciences Laboratory*

*Prepared for* COMMANDER SPACE SYSTEMS DIVISION  
UNITED STATES AIR FORCE  
*Inglewood, California*



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Report No.  
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A PORTFOLIO OF EXPERIENCE IN  
REFRACTORY METAL PROTECTIVE SYSTEMS RESEARCH

Prepared by  
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## ABSTRACT

This report supersedes Report TDR-169(3240-31) TR-1, "Re-Entry Protective Systems: A Bibliography of Refractory Metals Protective Systems Research," published 6 August 1962. The purpose of the present document is twofold: to direct research in the field of refractory metals protective coatings away from present silicide approaches and into systems capable of protection over 3500°F, and to familiarize the materials engineer with past and current research in his field (including the valuable examples set by silicide protection theory), thereby enabling him to interpret his own science for the designer. The report is divided into five major sections: molybdenum (366 references); columbium (299 references); tungsten (497 references); tantalum (229 references); and a new section, general (2401 references), which contains references to oxidation testing of refractory metal systems and to areas of research allied to the one vital problem, the protection of these metals from unfavorable environments at elevated temperatures.

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## PREFACE

### A. GENERAL

This report supersedes Report TDR-169(3240-31)TR-1, "Re-Entry Protective Systems—A Bibliography of Refractory Metals Protective Systems Research," which was compiled by the author and published on 6 August 1962. That report contained 2014 references and was split into four sections: molybdenum (739 references); columbium (226 references); tungsten (219 references); and tantalum (828 references). The present report, although fully inclusive of its precursor, is assembled somewhat differently. A new section, "General," has been added which contains all references on oxidation testing of refractory metal systems as well as on areas allied to the one vital problem—the protection of these metals from unfavorable environments at temperatures above 3500°F. As in the first report, these general references (now collected rather than scattered among molybdenum, tantalum and the others) include: meteorite impact considerations; the effects of vacuum noise, and coating-impurities-interstitial-with-the-substrate on the properties of the substrate; the effects of radiation and emittance coatings on protective coatings; impregnants on protective coatings; and coatings on substrates. Among test procedures referenced are analytical processes for finding impurities, methods for evaluating coated structures, and the simulation of environments individually and collectively. Surface response phenomenon on exposure to nuclear radiation is one indicative example which was recently extracted from the Russian literature on refractory metals. No attempt has been made to include references to research on the physical properties of specific metals unless it pertains to the subsistence of external protective systems, e. g., vapor pressure and thermal expansion data.

As in the previous document, entries are arranged alphabetically by author within each section; publications of unknown authorship are arranged at the end of each section alphabetically by title, where possible. All entries are consecutively numbered throughout the entire volume.

## B. DISCUSSION

This volume is not a bibliography in the ordinary sense of the word. The material it contains was not assembled by a library survey, but was chosen on the basis of subject matter from the author's files and from the bibliographies of company-issued reports. Certain of the works cited will require some research and effort to obtain, as approximately forty per cent of the material included has not been formally published and will not be readily accessible through technical libraries. Why such material has been included can best be explained by a statement of the basic purpose of the document: it was compiled as a source book of experience which can indicate new directions for research. Although it can also serve as a bibliography, it was intended to be read as if it were a research report.

## C. CONCLUSIONS

Examples of some references which the reader may wish to follow up are certain Russian works which pertain mineralogically to the extraction of refractory metals from their ores, references which dealt with refractory metal halogen chemical reactions, organometallic and oxygen compound reactions. The existence of such varied basic research disciplines in the thrifty Russian economy underscores the basic precept of this bibliography: thorough acquaintance with subtly applicable basic research and applied research conducted in the realm of recent understanding is prerequisite to avoid duplication and to engage in new research. Thus, this compilation is not intended as a research book for the designer—but rather as a design manual for the researcher.

## D. ACKNOWLEDGMENTS

A number of references pertain to the Refractory Composites Working Group Meetings NASA/ASD (formerly titled HTIRC-High Temperature Inorganic Refractory Coatings Working Group Meeting WADD). This group, begun by D. Roller and now monitored by L. N. Hjelm, ASD, and J. Gaugler, NASA, is responsible for stimulating a great deal of research in the field of refractory metal protective systems.

In the last bibliography, a request was made for additional material. About a month after its issue a voluminous card file was received from D. Roller, Magna Corporation, Anaheim, California, and D. W. Gates, Research Projects Division, George C. Marshall Space Flight Center, NASA, Huntsville, Alabama, with 600 references which were an outgrowth of their "Refractory Coatings Bibliography," revised June 1962. This material is included herein. Also, R. A. Long of Narmco, San Diego, California, sent an excellent bibliography which he compiled for this report on the subject of silicides. Since the issuance of the last report many other fine bibliographies and compilations have been received from researchers in the field for which the author is grateful. Acknowledgment is made of the excellent bibliography by C. J. Wensrich of University of California's Lawrence Radiation Laboratory entitled "Molybdenum, Niobium, Tantalum, Tungsten and Uranium Oxide in the Journal Literatures of the USSR, 1955-June 1960" (Sept 1, 1960), executed under Contract No. W-7405-eng-48 and containing 32 pages of excellent references, many of which were incorporated herein. Acknowledgment is also made for the bibliography from C. A. Krier's "Coatings for the Protection of Refractory Metals from Oxidation" which is included herein, as well as the hundreds of other bibliographies in the reports from the files of G. Kendall, H. Conrad, and P. Stedry of Aerospace Corporation.

Acknowledgment is also made of the excellent work of the recently formed Aerospace Information Division of the Library of Congress, whose abstracts and translations of the Russian literature will make this compendium unique, and of the time spent by K. Trirogoff of the Aerospace Corporation Library Research Group in checking references to AID accessions. Acknowledgment is also made for a literature survey on tungsten conducted by Edith Moore of the Library Research Group, the results of which were made available to the author before their publication for inclusion in this volume.

Finally, acknowledgment is made for both this bibliography and the preceding one to the excellent editorial support of the writing and editing section of the Publications Group of Aerospace, and particularly to E. Stickel, for standardizing the format and checking the references for validity and completeness.

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R-O - Reviewed and categorized other than silicide approach.

R-P - Reviewed and categorized protective refractory metal structural system.

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R-S - Reviewed and categorized silicide approach to protection.

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R-P - Reviewed and categorized protective refractory metal structural system.

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<p>Aerospac Corporation, El Segundo, California. A PORTFOLIO OF EXPERIENCE IN REFRACTORY METAL PROTECTIVE SYSTEMS RESEARCH. Prepared by D. H. Leeds. 11 March 1963. [389]p. (Report TDR-169(3240-31)TR-2:8ED-TDR-43-51) (Contract AF 64(695)-169) Unclassified report</p> <p>This report supersedes Report TDR-169(3240-31) TR-1, "Re-Entry Protective Systems: A Bibliography of Refractory Metals Protective Systems Research," published 6 August 1962. The purpose of the present document is twofold: to direct research in the field of refractory metals protective coatings away from present silicide approaches and into systems capable of protection over 3500° F, and to familiarize the materials engineer with past and current research in his field (including the valuable examples set by silicide protection theory), thereby enabling him to interpret his own science for the designer. The report is divided (over)</p>	<p>UNCLASSIFIED</p> <p>UNCLASSIFIED</p>
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